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METHODS OF TEST FOR PRINTING INKS

(Second Reprint JULY 1986)

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Indian Standard

METHODS OF TEST FOR PRINTING INKS

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CONTENTS

PAGE

0. FOREWORD 4
1. SCOPE 4
2. TERMINOLOGY 4
3. SAMPLING 5
4. TEST METHODS 5
    4.1 Hue (Shade) and Opacity 5
    4.2 Strength 6
    4.3 Relative Density/Density 7
    4.4 Viscosity 9
    4.5 Dispersion 14
    4.6 Printing Strength Covering Power and Distribution 17
    4.7 Strike Through and Show Through 20
    4.8 Tack 20
    4.9 Drying Time 22
    4.10 Setting Time on Coated Paper 23
    4.11 Coverage 24
    4.12 Solvent Extracted Residue 25
    4.13 Resistance Tests 25
        4.13.1 Resistance to Artificial Light 26
        4.13.2 Resistance to Water 26
        4.13.3 Resistance to Alkali 27
        4.13.4 Resistance to Acid 27
        4.13.5 Resistance to Alcohol 27
        4.13.6 Resistance to Solvents 27
        4.13.7 Resistance to Wax and Fat 28
        4.13.8 Resistance to Soap 28
        4.13.9 Resistance to Spices 28
    4.14 Resistance to Rubbing 29
    4.15 Resistance to Breakdown in Fountain Solution ... 30
    4.16 Lead (for Lead Restricted Inks) 31
    4.17 Ink Fly 32

APPENDIX A METHODS OF SAMPLING 32

Indian Standard

METHODS OF TEST FOR PRINTING INKS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 20 December 1972, after the draft finalized by the Printing Inks and Allied Products Sectional Committee had been approved by the Chemical Division Council.

0.2 The Printing Inks and Allied Products Sectional Committee having felt the need to prescribe agreed methods of test for printing inks decided to formulate this standard. While laying down the test procedures, it has been kept in mind to prescribe instruments which are available in the country except where it was found absolutely necessary.

0.2.1 Authentic data is not available for an objective assessment on some of the requirements independent of an approved/standard sample. Efforts to produce standardized procedure with instruments have not been successful. Comparative tests against a standard sample are accepted as giving the only reliable measure for such characteristics.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS:2-1960*.

1. SCOPE

1.1 This standard prescribes the methods of sampling and test for printing inks.

2. TERMINOLOGY

2.1 For the purposes of this standard, the definitions given in IS:4724-1968† in addition to the following shall apply.

2.1.1 *Approved/Standard Sample* — The sample accepted by the indentor or inspection authority as the basis for supply/manufacture.

---

*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to printing inks and allied industries.

3. SAMPLING

3.1 The method of drawing representative samples of the material shall be as prescribed in Appendix A.

4. TEST METHODS

4.1 Hue (Shade) and Opacity

4.1.0 Hue and opacity are compared with an approved/standard sample by drawdown test.

4.1.1 *Materials*

4.1.1.1 *Steel knife* — A slightly flexible steel knife whose width at the base is approximately 75 mm and whose edge has been carefully bored to a straight edge, free from sharp edges and burrs.

4.1.1.2 *Pad* — a firmly held pad of paper.

4.1.1.3 *Bond paper* — with a black bar approximately 20 mm wide (*see* IS : 1848-1971*).

4.1.2 *Procedure* — Place a sheet of bond paper having a black bar over a pad of paper. Put small quantities (about 0·2 ml) of each of the two inks to be compared 2 to 5 cm above the black bar and at a distance of about 2·5 cm from each other. Spread the inks by firmly scraping them towards the operator with a steel knife. At the start of the operation hold the blade nearly vertical, but with the handle slightly inclined towards the user and apply firm pressure. Towards the end of the stroke, release the pressure and bring the knife nearly parallel to the paper. The top of the drawdown shall consist of broadening streaks of the two inks, joining at the centre line, and of film thickness comparable with that of a printed film. The lower end of the drawdown shall be of a comparatively thicker film. Compare, both by reflected and transmitted light, the hues (shades) of the two inks on the top thin part of the drawdown.

4.1.2.1 The relative opacities of the standard ink and the sample shall be judged by the degree to which each hides the underlying black bar.

NOTE 1 — The two inks being compared should be of equal consistency. If they are not, films of unequal thickness will be drawn down, and will appear to have dissimilar hues.

NOTE 2 — The pad is used as a cushion to the drawdown paper so that the knife does not tear or damage the paper and yet firm pressure is obtained.

---

*Specification for writing and printing papers (*first revision*).

4.2 Strength

4.2.0 Differences in strength between two inks can be detected by observation of suitable drawdowns and prints, but it is not easy to make a quantitative estimation from such evidence. This is done by a 'reduction' test, where known mass of both approved/standard ink and sample ink are mixed with twenty times their own mass of opaque white ink.

4.2.1 *Apparatus*

4.2.1.1 *Glass slab* — suitable for ink mixing.

4.2.1.2 *Steel knives* — as in **4.1.1.1.**

4.2.1.3 *Glass slides*

4.2.2 *Reagent*

4.2.2.1 *Zinc oxide paste* — consisting of zinc oxide 70 parts and light stand oil (*see* IS:79-1950*) 30 parts.

4.2.3 *Procedure* — Place a known mass or volume (1 g or 1 ml) of ink on the glass slab. Add 20 g of zinc oxide white paste and mix the two intimately using the flexible palette knives until no streaks of white or colour are present. Treat the approved/standard sample in a similar manner. Compare small quantities of the resultant tints by placing them side by side on the bond paper and make a drawdown as prescribed in **4.1.2.** If they vary in strength, add known amounts of zinc oxide paste to the stronger tint until a match is obtained. The strength or weakness of the sample is expressed as a percentage of the approved sample.

4.2.3.1 In the case of white inks and extenders, a corresponding test is carried out by adding known amounts of a coloured ink to both.

4.2.4 *Calculation* — Taking the standard strength as 100, calculate the strength of a test sample of ink as follows:

$$\text{Relative strength} = \frac{M_2}{M_1} \times \frac{100}{1}$$

where

M_2 = mass in g of zinc oxide paste required for the test sample, and

M_1 = mass in g of zinc oxide paste required for standard ink.

If the test sample is weaker than the standard, the number denoting strength will be less than 100 and, if the test sample is stronger than the standard, the number will be more than 100. This number denotes the number of parts by mass of the test sample which are equivalent in strength to 100 parts by mass of the standard sample.

---

*Specification for stand oil for paints, (a) light, (b) medium, (c) heavy, (d) extra heavy.

4.3 Relative Density/Density

4.3.0 Three methods are recommended, namely, Method A, Method B, and Method C. Method A is suitable for highly viscous materials, Method B for less viscous materials, while Method C is suitable for rapid determinations.

4.3.1 *Method A*

4.3.1.1 *Principle* — The sample is weighed in air and water using the Archimedes principle.

4.3.1.2 *Procedure* — Suspend a metal plate about 25 mm^2 and 1 mm thick from a fine wire. Weigh it in air and again while immersed in water at 27°C, as shown in Fig. 1.

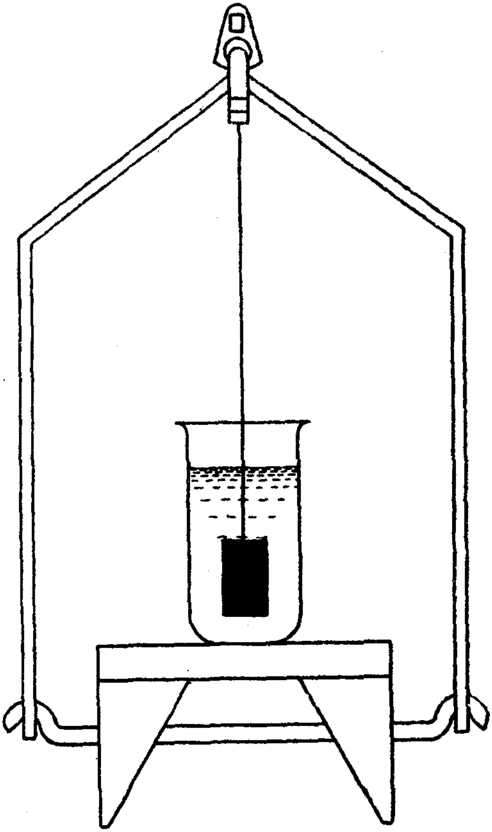


FIG. 1 MEASUREMENT OF RELATIVE DENSITY OF INK

Dry the plate, coat with about 1 g of ink and reweigh in air and in water. Care shall be taken not to incorporate air bubbles when coating the metal plate and to see that any air bubbles clinging to the surface are dislodged before weighing it in water. A small percentage of a wetting agent may be added in water.

4.3.1.3 *Calculation*

$$\text{Relative density} = \frac{M_1 - M_2}{(M_1 - M_2) - (M_3 - M_4)}$$

where

M_1 = mass in g of plate and ink in air,
M_2 = mass in g of plate in air,
M_3 = mass in g of plate and ink in water, and
M_4 = mass in g of plate in water.

4.3.2 *Method B*

4.3.2.1 *Apparatus* — Hubbard relative density bottle (modified), consisting of a straight walled bottle 40 ± 2 mm in diameter at the base, 22 ± 2 mm in diameter at the open end and 70 ± 2 mm in height, provided with a solid ground glass stopper with central bore of 1·6 ± 0·2 mm in diameter in place of the usual capillary opening. The lower part of the stopper is made concave with the depth of the cup-shape depression of 4·8 mm to allow all air bubbles to escape through the bore. The total mass of the bottle shall be not more than 35 g.

4.3.2.2 *Procedure* — Clean, dry and weigh the bottle and the stopper. Fill with distilled water at a temperature of about 23°C, introduce the stopper and keep it in a water bath at 27°C for 15 minutes. Wipe off all surplus moisture from the surface with a soft, clean and dry cloth and weigh again. Empty the bottle clean and dry it, and repeat the operations with the material.

4.3.2.3 *Calculation*

$$\text{Density} = \frac{c - a}{b - a}$$

where

c = mass in g of the bottle filled with the material,
a = mass in g of the dry bottle, and
b = mass in g of the bottle filled with water.

4.3.3 *Method C*

4.3.3.1 *Apparatus* — Ink pipette, as prescribed in **4.6.1.2**, and shown in Fig. 7.

4.3.3.2 *Procedure* — Fill the pipette with the ink by retracing the plunger and wipe off the end clean and level. Squeeze out a known volume of ink and cut off the resulting sausage with a weighed razor blade or a weighed piece of waxed paper. Again weigh the razor blade/ waxed paper and the ink.

4.3.3.3 *Calculation*

$$\text{Density} = \frac{M}{V}$$

where

M = mass in g of the ink squeezed out, and
V = volume in ml of ink.

4.4 Viscosity

4.4.0 For viscous inks falling bar viscometer is recommended, while for thinner inks like rotary newsprint ink, flow cup viscometer can also be used.

4.4.1 *Falling Bar Viscometer Method*

4.4.1.0 Printing inks are non-Newtonian substances possessing more or less definite yield values which must be overcome before flow is possible, and whose viscosities decrease with increasing rate of shear.

4.4.1.1 *Apparatus* — The falling bar viscometer shall be as shown in Fig. 2.

The instrument consists essentially of a cylindrical bar which is free to move inside a thick walled cylinder. The cylinder is seated in a water jacket and can be maintained at a constant temperature by circulating water through the jacket from a suitable constant temperature supply. The water jacket and supporting side rods are firmly fixed to a heavy base which is fitted with a spirit level and adjustable levelling screws. Weights are supplied which may be added to the top of the bar to increase the shearing force as required. Two types of bar and weights are available:

a) A steel bar with weights for loading in increments of 100 g up to a total of 1 000 g. This bar is intended for use with printing inks of high consistency.

b) A light alloy bar with weights up to a total of 100 g for use with inks of low consistency such as those used in the printing of polyethylene bottles.

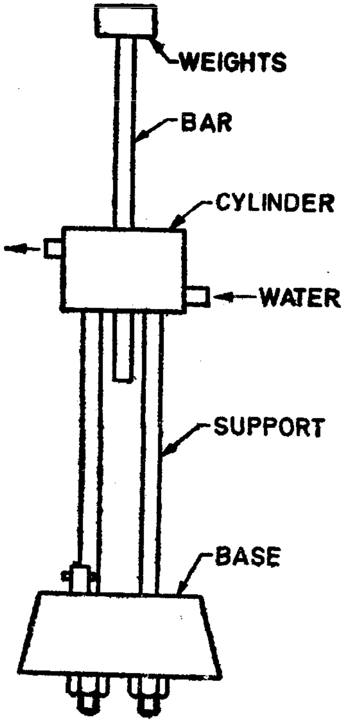


FIG. 2 FALLING BAR VISCOMETER

4.4.1.2 *Operating principle* — The viscometer bar is coated with the material under test and is allowed to fall through the viscometer cylinder so that the material is sheared between two concentric parallel surfaces. The fall of the bar is timed over a distance of 10 cm between two fixed marks on the supporting bars of the viscometer; this time is proportional to the viscosity of the material under test. By applying weights to the top of the bar and repeating the procedure a series of readings is obtained from which yield value and plastic viscosity may be derived.

4.4.1.3 *Procedure*

a) *Setting up the instrument* — The viscometer shall be screened from direct sunlight and from hot or cold draughts to avoid temperature variations during use. Place the cylinder in position and make sure the instrument is horizontal by adjusting the screws at the feet of the instrument and checking the spirit level on the instrument base.

Bring the viscometer cylinder to temperature of 27 ± 1°C by circulating water from the controlled water-bath. If the ambient temperature differs very widely from 27°C the sample to be tested shall be conditioned by storing in an oven at 27°C for at least 12 hours.

b) *Viscosity measurements* — Take about 1 ml of ink with the end of a spatula and make a circular coating on the bar 5 cm from one end. Thrust the bar downwards through the cylinder with a rotating motion to ensure that the bar is covered with ink. Hold the cylinder in position with one hand and raise the bar until its lower end is in level with the top mark. Apply a further 1 ml of ink to the cavity which appears where the bar meets the cylinder and rotate the bar to ensure uniform distribution. Allow the bar to fall under its own mass and note the time taken for the bar to pass from the middle to the lowest graduations on the side supports.

When timing with the aid of a stop-watch, the eye shall be in level with the graduation marks being observed. If an automatic timer is used the rate of fall of the bar is automatically measured and registered on a digital counter.

Repeat the operation placing weights on top of the bar to increase the shearing force. Between each repeat reading, the thick ring of ink which will be found at the top of the bar when it is raised shall be transferred by means of a spatula to the base of the bar.

4.4.1.4 *Recording of results*

a) When timing with a stop watch, readings taking longer than 300 seconds or less than 3 seconds are rejected. When using an automatic timer, time of fall down to 1 second can be recorded. In all cases at least four readings at different loadings shall be recorded.

b) The results in terms of added load and time of fall are plotted on a chart, and a straight line is drawn touching through the maximum number of points. Extrapolation of this line until it cuts the K_0^2 axis will give the yield value in dynes/cm². A second line is drawn parallel to the first but passing through the left-hand origin on the K_0^2 axis. Extrapolation of this line until it cuts the right-hand axis or top axis will give the plastic viscosity in poises.

c) A typical chart for use with a stop-watch and steel bar is shown in Fig. 3. Points have been plotted and joined by a continuous line to cut the K_0^2 axis at an yield value of about 10 000 dynes/cm² axis. The second line drawn parallel to the first and denoted in the diagram by a broken line cuts the plastic viscosity axis at about 170 poises.

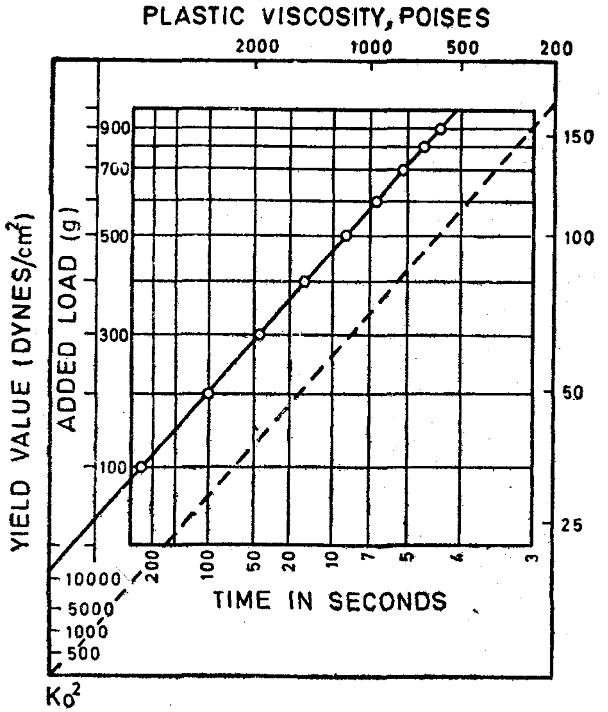


FIG. 3 VISCOSITY CHART FOR FALLING BAR VISCOMETER

4.4.1.5 *Precautions* — The following precautions shall be observed:

a) The test sample should be free of any dried skin particles which impede the fall of the rod.

b) The instrument shall be cleaned with white spirit or, preferably, acetone. Care should be taken not to scratch the surface of the rod and the inner face of the cup.

c) It is important that the instrument should at all the times be horizontal. The spirit level should be checked whenever the position of the instrument is altered. The outside of the cylinder and the inside of the water-jacket should be kept free of dried ink to ensure the proper seating of the cylinder.

d) Weights should be added to the bar in an ascending order of magnitude in suitable increments and not in random fashion.

e) When taking readings at temperatures which differ widely from the ambient temperature sufficient time should be given for the ink and bar to reach the temperature of the viscometer cylinder. This process may be speeded up by coating the bar with ink and allowing it to fall through the cylinder once or twice before the first time of fall is recorded. Every precaution should be taken to ensure that the temperature of the samples does not change during measurement. Viscometer should be used in conjunction with a temperature controlled water supply which may be circulated through the water jacket.

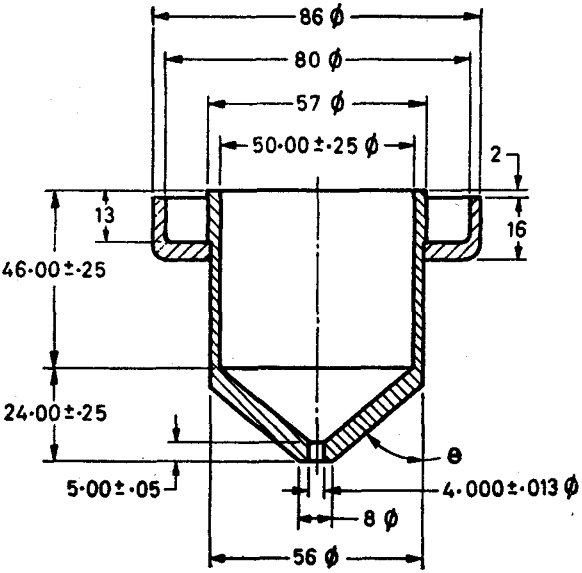
f) All surfaces should be perfectly clean and dry before the viscometer is used again.

g) When a thermostatically controlled water-bath is not available the instrument may be used to determine the yield value and plastic viscosity of a sample at the ambient temperature. In this case the temperature of the test sample and the air temperature should be recorded.

4.4.2 *Flow Cup Method*

4.4.2.1 *Apparatus*

a) The flow cup* shall be essentially of the form and dimensions as shown in Fig. 4.



All dimensions in millimetres.

FIG. 4 FLOW CUP

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*Generally known as Ford cup viscometer No. 4.

b) *Material of construction* — A cup made of any non-ferrous material is suitable. This may be plated. The finish shall be smooth. The jet may be either bored directly or constructed separately of stainless steel and force fitted. Care is essential in order to avoid damage to the lower apex of the cup. A protective skirt which does not interfere with the flow may be provided.

c) The following additional apparatus shall also be required in carrying out the test:

1) A thermometer accurate to within 0·5°C;
2) A stop-watch or stop-clock;
3) A suitable stand, provided with levelling screws;
4) A spirit level; and
5) A straight-edged scraper for the top of the cup.

4.4.2.2 *Procedure* — Place the flow cup on the stand in a place free from draughts, preferably with the air temperature within the range of 27 ± 2°C and level by using of a spirit level placed on the rim.

a) Strain the sample into a clean container and adjust the temperature to meet the requirements as specified in **4.2.2.2** (c). A 150-micron IS Sieve (*see* IS : 460-1962*) or finer, is suitable. This and the following operations shall be carried out with minimum delay to avoid loss of solvent.

b) With the orifice closed by the finger, fill the cup with the bubble-free sample until it just begins to overflow into the gallery, pouring slowly to minimize the formation of air bubbles. If bubbles are present, allow them to rise and then remove them from the surface.

c) Check that the temperature of the material in the cup is within 0·5°C of the test temperature. The cup may be at a temperature different from that of the sample and it is recommended that a minute or so be allowed to elapse before checking the temperature.

d) Place the scraper on the rim of the cup and draw it firmly across until the excess of the sample has flowed into the gallery. Place the receiver under the cup. Remove the finger and simultaneously start the stop-watch. Watch the stream of liquid flowing from the orifice. At the first evidence of a break of the stream into droplets, stop the stop-watch. The time taken is recorded in seconds as time of flow in the flow cup.

4.5 Dispersion

4.5.0 Dispersion is measured on a 'fineness of grind gauge' consisting of a block of hardened steel into which has been machined a shallow groove varying in depth from zero at one end to 0·102 mm at the other. A sample of ink is placed at the 'deep' end and is drawn down towards the shallow end by a steel scraper. Observation of any scratches in the ink film, and where they occur in terms of distance along the gauge, gives a measure of the degree of dispersion in the ink sample.

---

*Specification for test sieves (*revised*).

4.5.1 *Apparatus*

a) *Gauge** — a hardened steel block approximately 180 mm in length, 63·5 mm in width and 12·5 mm in thickness. The top surface of the block shall be ground smooth and flat, and shall contain a groove 133·4 mm in length and 12·5 mm in width, centred in the top of the block. The groove shall be tapered uniformly in depth length-wise from 0·102 mm at one end to zero depth at the other, calibrated with a scale number in accordance with its depth as given in Table 1.

NOTE — The apparatus may also be of a double channel type for comparing another sample.

b) *Scraper* — a double-edged steel blade 87·5 mm long, 37·5 mm wide and 6·8 mm thick. The two edges along the length shall be rounded to a radius of approximately 0·25 mm.

TABLE 1 RELATIONSHIP BETWEEN DEPTH OF TAPERED GROOVE AND SCALE FOR FINENESS OF GRIND

DISTANCE FROM ZERO END, mm	DEPTH, Microns	HEGMAN SCALE
133·350	102	Well for sample
127·000	102	0
111·125	89	1
95·250	76	2
79·375	64	3
63·500	51	4
47·625	38	5
31·750	25	6
15·875	13	7
0	0	8

4.5.2 *Procedure* — This consists of the following steps:

a) Place the gauge on a flat, non-slippery surface, and wipe it clean immediately before the test.

b) Place the material to be tested in the deep end of the groove so that it overflows the groove slightly. Care shall be taken to see that the sample is free of air bubbles.

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*Generally known as Hegman gauge.

c) Using both hands, hold the blade perpendicular to the block surface and at right angles to the length of the groove, draw the material down the length of the groove with a uniform deliberate motion. Use sufficient pressure to clean the level face of the gauge.

d) Immediately read the fineness as follows:

   i) View the gauge from the side so that the line of vision is at right angles to the longer dimension of the groove.

   ii) Hold the gauge in light that will make the pattern readily visible.

   iii) For actual reading, make the angle between the face of the gauge and the line of vision not more than 30° nor less than 20°.

   iv) Normally scratches will appear on the surface of the ink due to agglomerates of pigment particles. Note the scale interval in which 3 or more scratches commence and record to the nearest scale division.

Using a fresh sample of the material each time, obtain three readings in like manner. The first drawdown and reading is preliminary in order to establish proper conditions and to locate the fineness pattern. With this known, the second and third readings can be made with a minimum time lapse between completion of drawdown and actual reading. No reading shall be considered for reporting fineness when the time lapse exceeds 10 seconds.

NOTE — In the case of highly viscous inks, a little medium shall be used for bringing the material to workable consistency.

4.5.3 *Care of Gauge*

a) The gauge shall be immediately cleaned after each reading. Use a solvent and a soft cloth. Keep the gauge covered at all times when not in use. Gauges that lie idle for extended periods of time shall be protected from rust.

b) Do not allow any hard material to come in contact with the gauge surface or scraper in any manner that might result in scarring or nicking. Tapping or scratching with other metallic surfaces shall also be avoided.

c) The scraper edge may be rendered unsatisfactory for use by wear of the contact edge or by warpage.

NOTE — Wear or warpage of the scraper may be noted by facing the edge of the scraper down on the smooth level face of the gauge and then inspecting the contact edge by means of a strong light, placed behind the gauge. Rocking the scraper forward or backward will reveal poor contact due to wear or warpage. If any face shows that the scraper has been damaged, it shall not be used.

4.6 Printing Strength Covering Power and Distribution

4.6.0 Two or three prints of the ink under test are taken on a specified paper. They are allowed to dry for 24 hours and examined for printing strength, covering power and distribution of the ink.

4.6.1 *Apparatus*

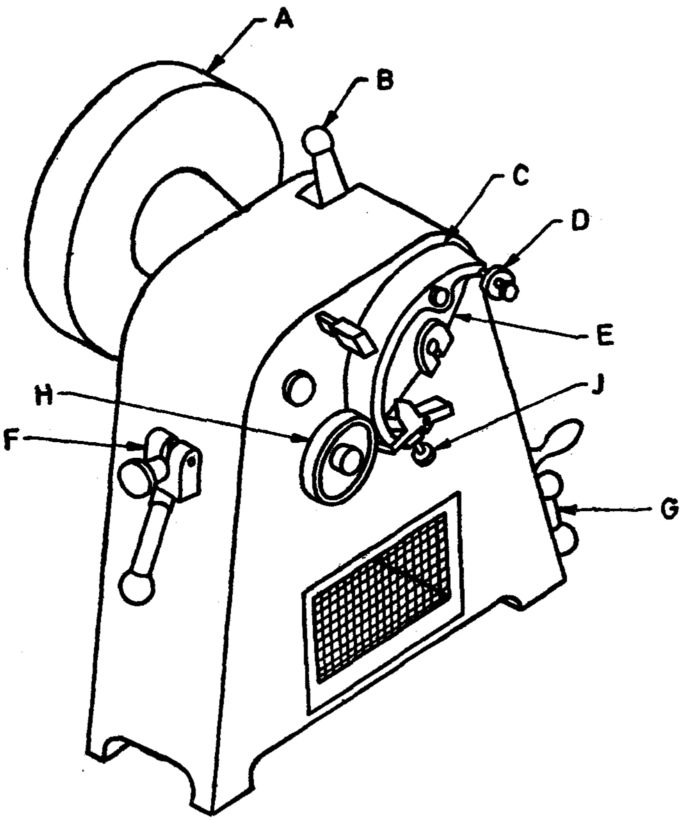
4.6.1.1 *Printability tester* — as shown in Fig. 5. The instrument is designed for taking a standard print. All the printing conditions, such as printing pressure, speed and quantity of ink used can be measured and controlled. The printing forme is represented by a circular disc *H* of either 1 cm or 2 cm in width. There is a motor driven inking mechanism which distributes a measured quantity of the ink uniformly and transfers it to the detachable printing disc.

The inking mechanism (*see* Fig. 6) consists of two inking systems making it possible to work simultaneously with two different inks and to quicken the test. Each inking system comprises of two steel rollers *M* and a synthetic composition roller *L* (forme roller). A measured quantity of ink is put on the forme roller *L* by means of ink pipette. Af er this the instrument is allowed to distribute the ink, so that it is uniformly spread out over the two steel rollers *M* and the forme roller. Then, the printing forme *H* is placed on the forme roller in order to be inked for a certain time as well. The thickness of the ink in the printing forme is in this manner kept constant to within 0·1 micron.

The printing forme *H* is then placed in the printing apparatus which provides the counterpart of an impression cylinder *E* in the shape of a section of 150°. The sector is covered with packing *C* or an offset blanket. On the other side of the apparatus, the spindle to which the sector is attached carries a weighted pendulum *A*. When released by a lever *B*, the pendulum descends with increasing speed. The print is produced by pressing the lever which releases the pendulum and causes the segment to rotate against the inked roller.

NOTE — The instrument is designed by the Institute of Graphic Art (IGT), Amsterdam, Holland.

4.6.1.2 *Ink pipette* — as shown in Fig. 7. This is a small pump made of thick-walled brass tube with a screw gauge. The ink is filled into the cylinder by opening the cap. The ink is extracted through a small hole in the plastic cap at the mouth of the cylinder by pressing the plunger in with the screw. The screw is calibrated to give the volume of ink extracted. On the outside of the tube, ten marks are engraved corresponding to the pitch of the screw thread.



LEGENDS

A *Weighed Pendulum* — By releasing it from starting position it serves to increase the printing velocity. It may also be operated by hand, producing approximately a constant velocity.

B *Lever* — This keeps the weight *A* and sector *E* in the starting position; by releasing it, both are allowed to fall.

C *Packing*

D *Tensioning Bar* — With this the packing *C* is brought under tension.

E *Sector* — This functions as the printing cylinder and can be fitted with a packing *C* and the paper to be printed.

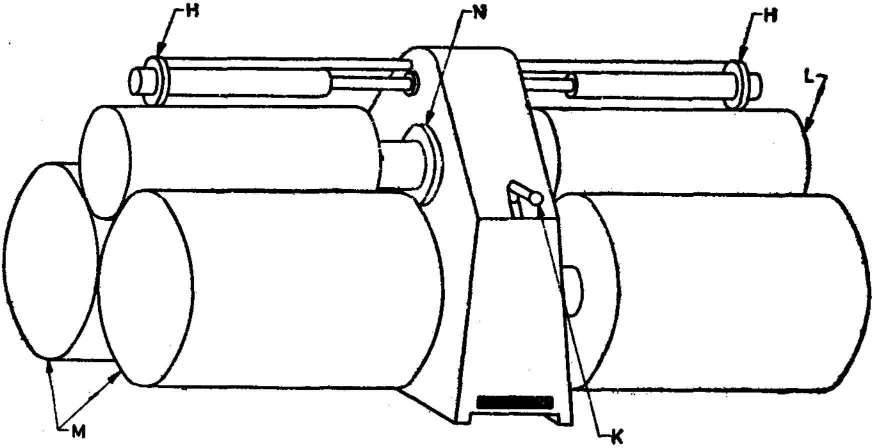
F *Packing Clamp*

G *Crank* — It serves to tension the spring which presses the printing disc *H* against the packing *C*. The spring tension is read on the diagram at the front.

H *Printing Disc* — This functions as printing forme and is inked up on the inking mechanism.

J *Clamp* — With this, the printing disc can be lifted from the packing *C*.

FIG. 5 PRINTABILITY TESTER



H *Printing Disc* — This functions as printing forme and is inked up on the inking mechanism.

K *Operating Handle of the Inking Mechanism* — In the upper position only the rear cylinders rotate. The rollers *L* are not in contact with the metal cylinder. In the middle position the instrument is set at rest. In the lower position the rollers *L* are in contact with the steel cylinder. Only the rear cylinders are driven by the motor and so the rotating of all cylinders is accomplished.

L *Forme Rollers* — Polyurethane or rubber rollers for working up water-based inks and moisture-set inks; glue-glycerine rollers for the use of oil-based inks.

M *Locking Ring* — Preventing the roller *L* leaving the instrument.

N *Thrust Spring* — By which the roller *L* is pushed slightly aside and the distribution is improved.

FIG. 6 INKING MECHANISM

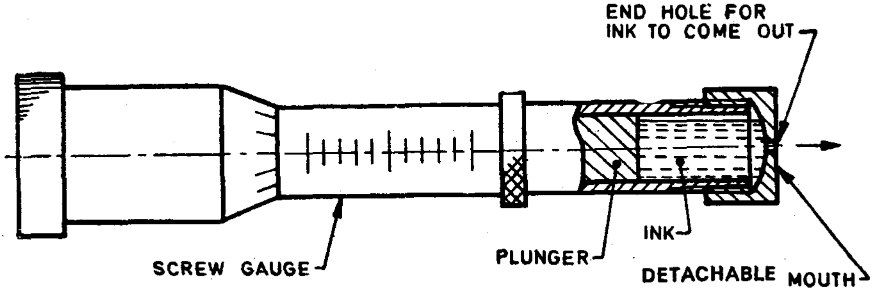


FIG. 7 INK PIPETTE

4.6.2 *Procedure*

4.6.2.1 Measure 0·75 ml of the ink by means of ink pipette and transfer it to the inking mechanism of the printability tester. Allow the ink to distribute for a minimum of 8 minutes and then ink thoroughly the inking disc. After this, attach the disc to the printability tester and adjust the pressure to 35 kg. Take a number of prints specified in the relevant specification of the ink on machine coated art paper (*see* Sl No. 12 of IS : 1848-1971*) at a constant speed of 20 cm per second and allow to dry for 24 hours. The procedure for producing prints at constant speed is as follows:

> Hold the weighted pendulum *A* firmly in the right-hand. Pull over the lever *B* with the left-hand leading the paper also in the left-hand so that the strip is not drawn aslant. The pulling over of the lever shall be completed in just one second resulting in a velocity of 20 cm per second. Remove the printed strip by releasing clamp *J*.

4.6.2.2 Examine the dried prints for uniformity of colour and whether any white areas or specks show through the dried film of the ink when prints are taken from solid disc or half tone disc as the case may be.

4.7 Strike Through and Show Through

4.7.1 Examine the reverse side of the print as prepared in **4.6.2.1** for stains and spots. The ink shall be considered to be not striking through or showing through the paper if there are no oily stains or impressions of ink on the back side of the paper and the printing is not visible through the sheet from the reverse side as a shadow image.

4.8 Tack

4.8.1 *Apparatus*

4.8.1.1 *Inkometer* — The instrument shall be as shown in Fig. 8.

The instrument consists of a metal roller, *X*, and a composition roller, *Y*. The latter is supported by and permitted to swing freely on the shaft of the lower roller. All bearings are practically frictionless. The lower roller is driven by an electric motor of 1/8 horse power capable of three different speeds, 200, 400 and 600 rpm. The upper roller is driven only by contact with the lower roller against which it rests. The upper roller thus functions as a brake on the lower one. A graduated balance arm *L* with a sliding weight *W* is attached to the frame in such a way that the torque acting on the steel frame may by balanced by moving the weight along the balance arm. The frame and the roller are so balanced that

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*Specification for writing and printing papers (*first revision*).

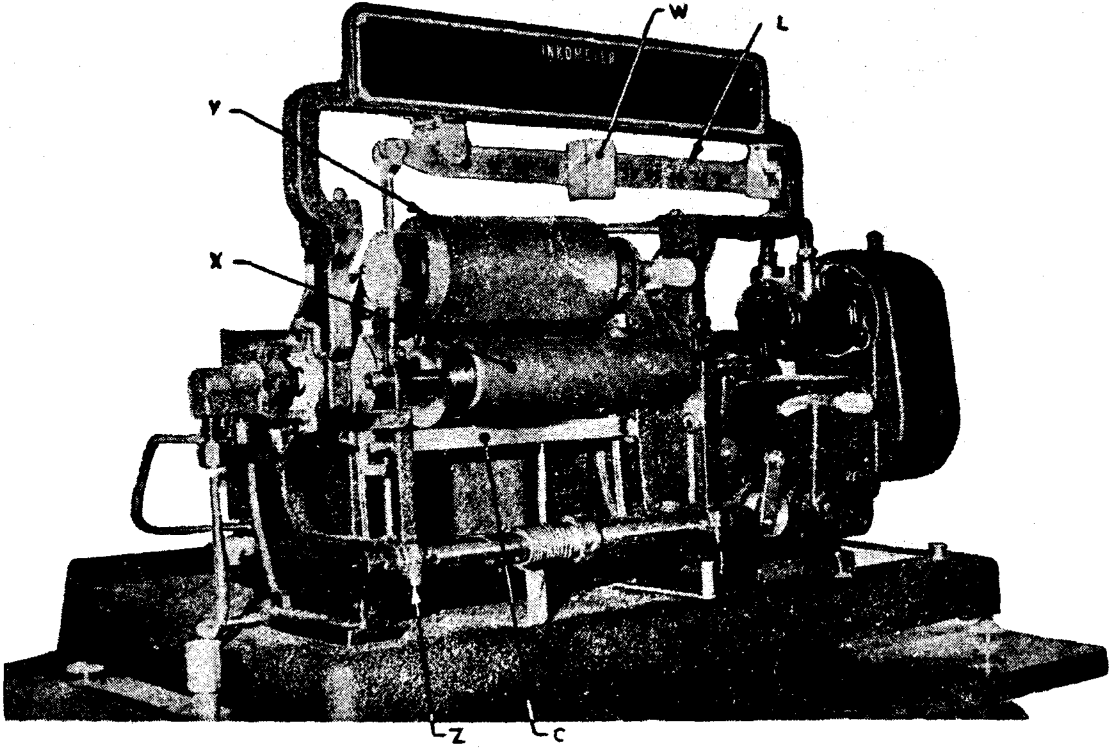

FIG. 8 INKOMETER FOR DETERMINATION OF TACK

the centre of gravity of the steel frame and upper roller is a little below the axis of the lower roller. This arrangement forms a dynamometer capable of indicating the frictional torque between the rollers at their point of contact. The friction, it may be noted, is found to be virtually independent of the speed when no ink is present on the rollers. This frictional force is overcome by the side-arm *Z* which carries a counter weight. The torque produced by an ink film does change with speed of the rollers, and this force is balanced by the sliding weight *W*, thus permitting measurement of tack of the ink under actual operating conditions.

4.8.2 *Procedure* — Keep the roller system, which is temperature controlled by a water circulating device, at 27°C. Ensure the machine is reading zero tack at the lowest speed with no ink on the rollers.

NOTE — At higher speeds with no ink on the rollers a 'false' reading will be given which is normally 1 unit at 800 and 2 units at 1 200. It is essential to determine this 'false' reading exactly before making a test at the higher speeds and subtract this factor from the scale reading.

4.8.2.1 Place from an ink pipette, 1·3 ml of ink upon the rollers. The ink shall be applied in a uniform fashion so as to avoid uneven distribution. Set the machine at 800 rpm and switch on. Run for 30 seconds to allow full distribution. Release the stop lever which allows the roller *Y* to swing round as far as the tack of the ink will pull it. Counter balance the swing with the weight by sliding it along the graduated arm until it goes down to the reference point and read the figure from this point on the arm. Subtract the factor necessary at the speed chosen (*see* Note under **4.8.2**) and the result is the 'tack' figure. The reading is accurate to ± 0·2 of a unit.

NOTE — The instrument mentioned in **4.8** is the Reed Inkometer manufactured by Thwing — Albert Instrument Company, UK. Other instruments, namely, Churchill/Metal Box Tackmeter or Patra Tackmeter can also be used for measurement of tack.

4.9 Drying Time

4.9.0 Drying time on machine is determined by rolling films of ink on glass, while drying time on paper is determined at letterpress thickness on drying time instrument.

4.9.1 *Apparatus*

4.9.1.1 *Ink pipette* — same as in **4.6.1.2.**

4.9.1.2 *Glass slab* — about 25 × 25 cm.

4.9.1.3 *Hand inking roller* — as shown in Fig. 9. It is about 20 cm in length and 5 cm in diameter, made from polyurethane or any reasonably hard synthetic rubber which is not likely to absorb solvents from the ink and cause swelling.

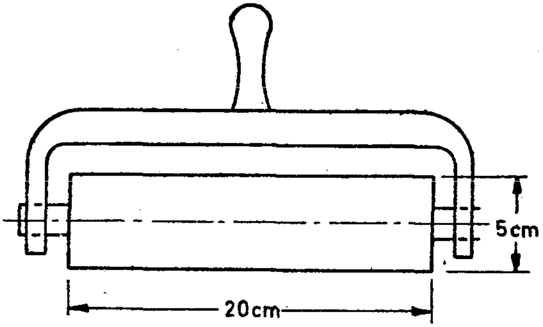

FIG. 9 HAND INKING ROLLER

4.9.2 *Procedure*—Place 0·5 ml of ink measured from ink pipette on a glass slab of approximately 25 × 25 cm size, and with the aid of hand inking roller, spread out the ink into a uniform film. The film thickness of ink will simulate the ink film on the rollers of a normal letter press machine. In one corner, place a blot of ink about 2 ml to simulate the ink in the ink duct. At known intervals of time draw a finger across the thin film. The ink shall be considered dry at the moment it does not smear. A pin drawn across the blot will indicate the same fact as a skin will be formed when the film dries. Note the time taken for the film to dry. The test shall be carried out at 27 ± 2°C and a relative humidity of 65 ± 2 percent.

4.10 Setting Time on Coated Paper

4.10.1 *Apparatus*

4.10.1.1 *Printability tester*—same as in **4.6.1.1.**

4.10.1.2 *Ink pipette*—same as in **4.6.1.2.**

4.10.2 *Procedure*—Measure 0·6 ml of the ink from the ink pipette and apply it to the ink distributor of the printability tester where it is distributed for ten minutes. The ink disc takes the ink from the roller for a period of exactly 45 seconds. Place a strip of machine coated art paper (*see* Sl No. 12 of IS : 1848-1971*) on which the print is to be made on the segment of the tester, apply pressure of 40 kg/cm² between the disc and the segment and take out a print.

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*Specification for writing and printing papers (*first revision*).

Immediately after the printed slip has come from the printability tester, place a blank slip of the same paper in contact with the printed side. After one minute, pass both the slips through the printability tester together with a blank disc, under a pressure of 5 kg/cm^2. Carry out this operation in three phases at intervals of one minute, so that on a slip the drying state is visible after 1, 2 and 3 minutes. The setting time is expressed as number of minutes at which there is no overprint on the blank slip.

The test shall be carried out at 27 ± 2°C and relative humidity of 65 ± 2 percent.

4.11 Coverage

4.11.0 This refers to the quantity of ink required to print a given area of a particular paper and is of commercial importance both to printers and ink makers.

4.11.1 *Procedure* — Take prints on the machine for which the ink is intended to be used and calculate the coverage as follows:

$$\text{Quantity of ink (in kg) required} = \frac{N \times A \times K \times P \times S \times G}{550\ 000}$$

where

N = number of one sides printed,

A = area of forme in cm^2,

K = kind of forme — *see* factors below:

Solids	*Reverse Lettering*	*Heavy Plate*	*Catalogue with Illustrations*	*Heavy Type*	*Light Type*
1·0	0·70	0·40	0·30	0·20	0·15

P = printing process — *see* factors below:

Letterpress	*Direct Litho*	*Offset*
1·0	1·0	0·50

S = stock — *see* factors below:

Art	*Imitation*	*Super Calender*	*Smooth Cartridge*	*News*	*Carton*	*Antique*	*Rough Cartridge*
1·0	1·2	1·3	1·6	1·8	2·0	2·0	2·2

G = relative density of ink as calculated in **4.3**.

4.11.1.1 *Example*—For 11 000 sheets, one side printing, 40 × 60 cm, by letterpress, in heavy type and display in royal blue having specific gravity of 1·4 on imitation art, the quantity of ink required is:

$$\frac{2\,750 \times 40 \times 60 \times 0{\cdot}2 \times 1{\cdot}0 \times 1{\cdot}2 \times 1{\cdot}4}{550\,000} = 4{\cdot}0 \text{ kg}$$

4.12 Solvent Extracted Residue

4.12.1 *Reagents*

4.12.1.1 *Ether*—solvent grade (*see* IS : 336-1964*).

4.12.1.2 *Acetone*—*see* IS : 170-1966†.

4.12.1.3 *Toluene*—*see* IS : 1839-1961‡.

4.12.1.4 *Ferric chloride*—solid.

4.12.2 *Procedure*—Weigh accurately about 10 to 15 g of the ink into a glass screw top jar, and to this, add 10 ml each of ether, toluene and acetone, alongwith a crystal of ferric chloride. (Ferric chloride is added to inhibit the tendency to form a fine colloidal suspension and consequently it promotes a clearer separation.) Shake the ink until it is in solution and allow it to stand overnight. Centrifuge the separated matter and wash it twice with the mixed organic solvents consisting of ether, toluene and acetone in equal proportions by volume. Dry in an oven at a temperature of about 70°C and weigh till constant mass is obtained.

NOTE—The extracted material will consist of pigments contaminated with a little highly polymerized varnish which has separated from the vehicle.

4.12.3 *Calculation*

$$\text{Solvent extracted residue, percent by mass} = 100 \times \frac{M_1}{M}$$

where

M_1 = mass in g of the residue obtained, and
M = mass in g of the material taken for the test.

4.13 Resistance Tests

4.13.0 These tests are for end-use properties on printed specimen. Prints are taken on IGT printability tester as specified in **4.6** and tested for resistance to light, water, acid, alkali, soap and alcohol.

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*Specification for ether (*revised*).
†Specification for acetone (*first revision*).
‡Specification for toluene, reagent grade.

4.13.1 *Resistance to Artificial Light*

4.13.1.1 *Apparatus: Fadeometer* — It shall have the following provisions:

a) *Source of light* — A carbon arc composed of carbon electrodes and enclosed in a heat-resistant glass globe to produce a complete spectrum from 279 *m/u* to 12 000 *m/u*.

The arc should operate on a direct current of approximately 11 to 13A° or on alternating current of 50 to 60 c/s and approximately 13 to 17A°, with 110 to 140 V across the arc.

b) The test specimens are vertically hung in test racks on the inner wall of a cylinder (rotating or fixed type), concentric with the arc. The distance of the test specimen shall be approximately 250 mm from the arc. No part of the specimen shall be more than 100 mm above or below the horizontal plane passing through the centre of the arc when it is fitted with new electrodes.

c) The air inside the chamber is humidified by placing water in a trough or water soaked cotton wicks in the chamber. A fan or blower unit keeps a flow of fresh air over the specimens and through the chamber.

4.13.1.2 *Procedure* — Take a printed sheet as obtained in **4.13.0** and after drying for the period determined in **4.9.2** cut it into two portions. Place one of the pieces at a distance of 250 mm from the carbon arc lamp of the fadeometer. Expose it to the radiation for the specified period. Maintain the temperature of the circulating air at about 54°C during the test. Examine the specimen against the unexposed piece.

NOTE — The above test is comparative. Alternatively, direct assessment may be made against a Grey Scale.

4.13.2 *Resistance to Water*

4.13.2.1 *Procedure* — Immerse three thicknesses of filter paper (size 60 × 90 mm each) in distilled water and allow to drip so that they remain saturated. Place the saturated filter papers on a glass plate. Place the printed specimen with its printed side in contact with the top filter paper and cover with another glass plate. Place a weight of 1 kg over it and store in an atmosphere saturated with water vapour at a temperature of 27 ± 2°C. After 24 hours, remove the specimen and dry in free air for 4 hours or in an oven at a temperature of 40°C for 45 minutes and compare any fading of the colour with an unprocessed print.

Dry the sheets of filter paper in the same manner after light padding with blotting paper on the reverse side to remove the bulk of water. Compare the filter paper which was in contact with the print for staining against a sheet of the same paper that has been saturated with the same water, similarly dried but has not been in contact with the print.

4.13.3 *Resistance to Alkali*

4.13.3.1 *Reagent* — Sodium hydroxide solution — 20 g/l.

4.13.3.2 *Procedure* — Take a specimen of printed sheet as prepared in **4.13.0** and after allowing it to dry for the specified period, cut a portion of about 5 square centimetres. Using a sample dropper, place several spots of 2 drops each of sodium hydroxide solution on the print and the blank portion of the test specimen. Allow the spots to dry in air and examine for any fading or discoloration of the spotted portion of the specimen as compared with the blank.

4.13.4 *Resistance to Acid*

4.13.4.1 *Procedure* — Take a specimen of printed sheet as prepared in **4.13.0** and after allowing it to dry, for the specified period immerse a part of it in 5 percent (*v*/*v*) hydrochloric acid for 30 minutes. Take it out, wash with running water and allow to dry. Examine the prints and the white paper below the print and compare with the unwetted portion.

4.13.5 *Resistance to Alcohol*

4.13.5.1 *Procedure* — Take a specimen of printed sheet as prepared in **4.13.0** and after allowing it to dry, immerse a portion of it in ordinary denatured spirit (*see* IS : 324-1959*) for 30 minutes. Take it out, and after allowing to dry, examine the prints and the white paper below the prints and compare with the unwetted portion for any bleeding or fading of the colour.

4.13.6 *Resistance to Solvents*

4.13.6.1 *Procedure* — Take a specimen of printed sheet as prepared in **4.13.0** and after allowing it to dry for the specified period, immerse a portion of it in a mixture of equal proportions of ethyl alcohol (*see* IS : 321-1964†), toluene (*see* IS : 1839-1961‡), butyl acetate (*see* IS : 230-1972§) for 30 minutes. Take it out and allow to dry. Examine the prints and the white paper below the prints and compare with the unwetted portion for any bleeding or fading of the colour.

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*Specification for ordinary denatured spirit (*revised*).
†Specification for absolute alcohol (*revised*).
‡Specification for toluene, reagent grade.
§Specification for normal butyl acetate (*second revision*).

4.13.7 *Resistance to Wax and Fat*

4.13.7.1 *Procedure* — Take a specimen of printed sheet as prepared in **4.13.0** and allow it to dry for the specified period. Melt paraffin wax in an evaporating dish and maintain at 95 to 100°C by a steam bath. Dip a portion of the print for one minute. Take it out and allow to drain and cool. Examine the prints and the white paper below the print for any bleeding or fading of colour as compared with the unwetted portion.

Carry out the test in a similar manner with fat or lard at 95 to 100°C using a fresh specimen of printed sheet.

4.13.8 *Resistance to Soap*

4.13.8.1 *Reagent: Soap gel* — 20 g/l, prepared by dissolving white and neutral toilet soap in distilled water, warming and making up the loss due to evaporation. Allow the soap gel to set in shallow vessels such as thin lid or dish for at least 24 hours.

4.13.8.2 *Procedure* — Take about 3 × 2 cm specimen of the print as prepared in **4.13.0**, moisten it with distilled water and paste it on the soap gel with the printed side touching the soap gel. Keep it in a desiccator containing water. After 24 hours, peel off the print and examine for any bleeding or transfer of ink. It is sometimes difficult to assess bleed in presence of transference and vice versa. For this, scrape the surface of the soap jelly and observe the layer underneath.

NOTE — Moist soap can have a disintegrating effect on paper and paper coating and this results in an apparent transference which is not indicative of breakdown of ink film or inferior pigment.

4.13.9 *Resistance to Spices*

4.13.9.1 *Procedure* — Take a specimen of printed sheet as prepared in **4.13.0** and allow it to dry for the specified period. Place it in a petri dish of about 120 mm dia and 40 mm height, with the printed side upward and cover with a sheet of filter paper of about 90 × 60 mm. Cover the filter paper with at least 3 mm thick of the particular spice to be tested. Place a glass plate of about 90 × 60 mm on the whole and put a weight of 500 g over it. Close the dish with the cover and keep at a temperature of 27 ± 2°C.

Remove the specimen after 7 days and examine the print for any change in colour in comparison with an unprocessed print. Also examine the filter paper for any bleeding of the colour in comparison with a sheet of the same paper that has been treated with spice but has not been in contact with the print.

4.14 Resistance to Rubbing

4.14.0 *General*—The method is primarily suited to test printed carton board, but may be used for paper, foil and plastics if the samples are suitably backed. A 0·6 mm smooth white lined chipboard is satisfactory.

4.14.1 *Outline of the Method*—The sample of print to be tested is rubbed forwards and backwards in a straight line by a second sample of print which is of a fixed size and under a fixed load.

4.14.2 *Apparatus*—as shown in Fig. 10. The instrument consists essentially of (a) a base board measuring 450 × 150 mm on which the test sample is placed, print upwards, and (b) a block measuring 100 × 75 × 50 mm high to the base of which the second sample is attached, print downwards. The gross mass of the block is 625 g.

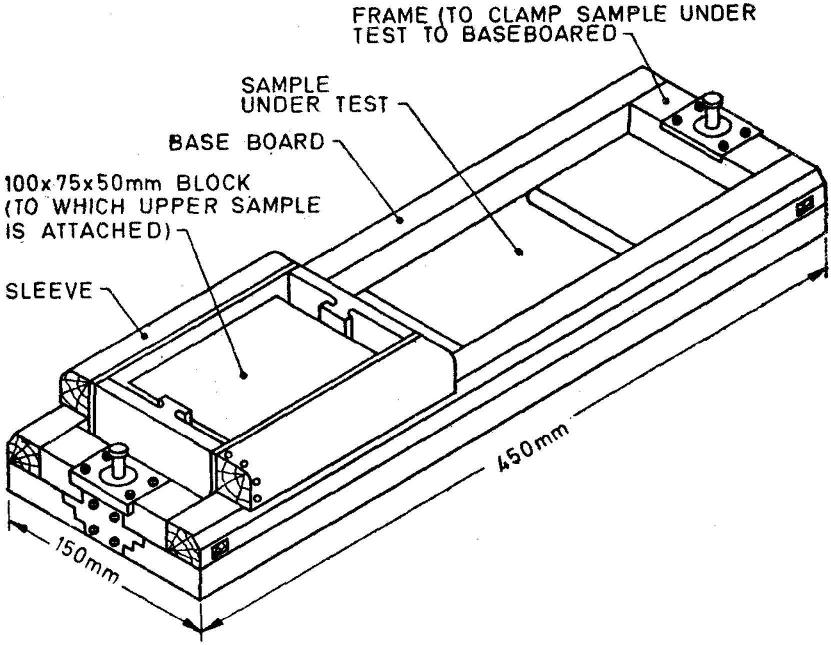
FRAME (TO CLAMP SAMPLE UNDER TEST TO BASEBOARED
SAMPLE UNDER TEST
BASE BOARD
100x75x50mm BLOCK (TO WHICH UPPER SAMPLE IS ATTACHED)
SLEEVE
450mm
150mm

FIG. 10 APPARATUS FOR TESTING RESISTANCE TO RUBBING

The whole instrument is made of hardwood except that the base of the block is formed from a brass plate 6 mm thick. This is screwed to the wood, the screw heads completely recessed below the surface of the metal.

The base of the block and the top of the baseboard shall be flat. A frame of 25 mm square section hardwood strip round the perimeter of the baseboard holds the test sample firmly. A sleeve slides along the sides of the frame without touching the baseboard. The block fits loosely inside this sleeve.

4.14.3 *Procedure* — Take a specimen of the printed sheet and cut a portion measuring 150 × 75 mm so that the longer dimension is parellel to the cross direction. Crease this about 25 mm from each end of the longer dimensions and place it, print outwards, on the base of the block. Use the end flaps to hold the sample firmly on the block. The test sample shall measure 150 mm wide in the machine direction. Place it, print upwards with the 150 mm dimension across the width of the baseboard and clamp it to the board by the frame. Carefully remove any dust from both samples with cotton wool.

4.14.3.1 Put pencil mark between 65 mm and 100 mm from each end of the longer dimension of the test sample. Put the block in position, with board sample downwards, at one end of the baseboard so that the whole space between the two pencil marks is exposed, fit the sleeve over the block. Apply force to this sleeve rather than to the block, so that the loading on the block is not accidentally increased by hand pressure. Move the block over the test sample until the pencil mark on the other end of the sample is uncovered. The movement shall be even at the rate of 30 cm per second. The movement forwards and back comprises one stroke.

4.14.3.2 Continue up to the specified number of stroke after which there shall be no signs of ink marking on the test sample.

4.15 Resistance to Breakdown in Fountain Solution

4.15.0 Breakdown of lithographic inks in the presence of fountain solution occurs primarily as a result of two separate mechanisms. The first which causes scumming, washing, bleeding, etc, is an oil-in-water emulsion formation which is responsible for the carrying of particles of ink to non-image areas on the plate. The second is excess water uptake producing water-in-oil emulsion that interferes with flow and distribution properties causing piling on the rollers in which state the ink is said to be water logged. Both these faults are detected by using a machine known as the 'lithobreak tester'.

4.15.1 *Apparatus*

4.15.1.1 *Lithobreak tester* — The apparatus shall be as shown in Fig. 11. The machine consists essentially of a small roller distribution chain of three rollers, provision being made for the comparison of two inks by a separation groove cut into the roller centres.

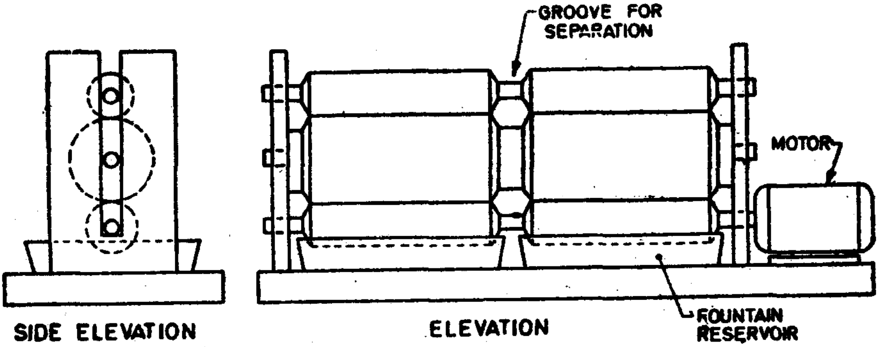

FIG. 11 LITHO BREAK TESTER

4.15.2 *Material*

4.15.2.1 *Fountain solution* — Acidic fountain solution of the following composition:

Phosphoric acid	0·2 g
Gum arabic	0·2 g
Distilled water (*see* IS : 1070-1960*)	99·6 g

4.15.3 *Procedure* — Apply 1·0 ml of the ink on the rollers and distribute it uniformly. Introduce the fountain solution in the reservoir and immerse one of the rollers in the reservoir. Rotate the rollers by means of the electric motor for a specified period and examine for any sign of oil and water emulsification in the fountain solution and for any piling or stripping on the rollers.

4.15.3.1 The test is repeated with distilled water in place of fountain solutions.

4.16 Lead (for Lead Restricted Inks)

4.16.1 *Procedure* — Transfer about 1 g accurately weighed residue obtained in **4.12.2**, to a 750-ml conical flask and add 500 ml of 2·5 percent (*v*/*v*) hydrochloric acid. Stopper it and shake mechanically for one hour. Allow the mixture to stand for one hour and then filter. Precipitate the lead salt contained in the clear filtrate as lead sulphide by passing a stream of hydrogen sulphide. Filter and heat the lead sulphide in air to

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*Specification for water, distilled quality (*revised*).

convert into lead sulphate. Weigh, calculate as lead monoxide (PbO) and express the result as percentage of the material taken for the test.

4.16.1.1 Alternatively, determine lead by electrolysis after dissolving the precipitated lead sulphide in concentrated nitric acid and calculate as lead monoxide (PbO).

4.17 Ink Fly

4.17.1 *Apparatus* — Inkometer (*see* **4.8.1.1**).

4.17.2 *Procedure* — Apply the ink under test to the rollers of the inkometer using the ink pipette and allow it to distribute for 1 minute at the speed specified in the relevant specification. Hold a folded sheet of paper (with the fold up) underneath the metal and composition rollers for one minute. Remove the paper, unfold it and examine the sheet. If flying has occurred, the area not covered during the test shall show a tint of the ink.

APPENDIX A

(*Clause* 3.1)

METHODS OF SAMPLING PRINTING INKS

A-1. GENERAL REQUIREMENTS OF SAMPLING

A-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

A-1.1 Samples shall not be taken in an exposed place.

A-1.2 The sampling instrument shall be clean and dry.

A-1.3 The material in the containers shall be mixed thoroughly before drawing samples.

A-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

A-1.5 The samples shall be placed in clean and dry glass containers.

A-1.6 After filling, the sample containers shall be closed tightly with a stopper, sealed and marked with full details of sampling, such as sample number, the date of sampling, the batch of manufacture of material, name of manufacturer and other important particulars of the consignment.

A-1.7 Samples shall be stored in such a manner that the conditions of storage do not unduly affect the quality of the material.

A-2. SCALE OF SAMPLING

A-2.1 Lot — All the containers of the same size in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, or of different sizes of containers belonging to the same batch and of the same size shall be grouped together and each such group shall constitute a separate lot.

A-2.1.1 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirements of the specification.

A-2.2 The number of containers (*n*) to be selected from a lot shall depend on the size of the lot (*N*) and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

LOT SIZE N (1)	NO. OF CONTAINERS TO BE SELECTED n (2)
Up to 20	3
21 to 40	4
41 ,, 80	5
81 ,, 120	6
121 ,, 200	8
201 and above	10

NOTE — In the case of very small lots where the selection of three containers may be uneconomical, the number of containers to be selected and the criterion for judging the conformity of the lot to the requirement of the specification shall be as agreed to between the purchaser and the supplier.

A-2.3 The containers shall be selected at random and in order to ensure the randomness of selection, a random number table shall be used (*see also* IS : 4905-1968*). In case such a table is not available, the following procedure may be adopted:

Starting from any container, count them in one order as 1, 2, 3,......, up to *r* and so on, where *r* is the integral part of N/n (*see* **A-2.2**). Every *r*th container thus counted shall be withdrawn to give samples for test.

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*Methods for random sampling.

A-3. PREPARATION OF TEST SAMPLES AND REFEREE SAMPLES

A-3.1 To ensure that the sample taken from each container is representative of the contents, the contents shall be mixed thoroughly by shaking or by stirring or both.

A-3.2 After the contents are thoroughly mixed, a small representative portion of the material shall be taken with the help of a suitable sampling instrument from each of the containers selected according to **A-2.3**.

A-3.3 In case it is not possible to attain a thorough mixing by shaking or stirring, small representative portions of the material shall be taken from different parts of the container with the help of a suitable sampling instrument so as to give representative sample for the container.

A-3.4 The material taken from each container shall be divided into three equal parts, each forming an individual sample. One set of the individual samples representing the *n* containers selected shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

A-3.5 All the samples shall be transferred to separate containers. These containers shall then be sealed air-tight and labelled with full identification particulars given in **A-1.6**.

A-3.6 The referee samples consisting of a set of *n* individual samples representing *n* containers selected shall bear the seals of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of any dispute between the two.

A-4. NUMBER OF TESTS

A-4.1 Tests for the determination of all the requirements prescribed in the relevant material specification shall be performed on each of the individual sample separately.

A-5. CRITERIA FOR CONFORMITY

A-5.1 A lot shall be declared as conforming to the requirements of the relevant material standard if the different test results as obtained under **A-4** meet the corresponding requirements given in the standard.

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